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Methyl 3-(4-methylbenzylidene)-carbazate

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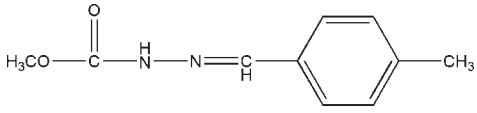
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.175; data-to-parameter ratio = 18.3.

The title compound, $C_{10}H_{12}N_2O_2$, was prepared by the reaction of methyl carbazate and 4-methylbenzaldehyde. The dihedral angle between the benzene ring and the carbazate fragment is 20.86 (10)°. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds.

Related literature

For background to Schiff bases, see: Cimerman *et al.* (1997). For C=N bond lengths, see: Girgis (2006). For a related structure, see: Li *et al.* (2009).



Experimental

Crystal data

$C_{10}H_{12}N_2O_2$ M_r = 192.22
Monoclinic, $P2_1/c$
a = 10.038 (2) Å
b = 13.308 (3) Å
c = 7.7923 (16) Å
β = 99.71 (3)°
V = 1026.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
9493 measured reflections
2322 independent reflections
1528 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.175
S = 1.06
2322 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.00	2.8615 (18)	176

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2684).

References

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Li, Y.-F., Liu, H.-X. & Jian, F.-F. (2009). *Acta Cryst.* E**65**, o2959.
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supporting information

Acta Cryst. (2010). E**66**, o1429 [https://doi.org/10.1107/S160053681001799X]

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S1. Comment

Schiff bases have received considerable attention in the literature. They are attractive from several points of view, such as the possibility of analytical application (Cimerman, *et al.*, 1997). As part of our search for new Schiff base compounds we synthesized the title compound (I), and describe its structure here.

The molcular structure of (I) is shown in Fig. 1. The C8—N2 bond length of 1.273 (2)Å is comparable with C—N double bond [1.281 (2) Å] reported (Girgis, 2006). In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds.

S2. Experimental

A mixture of methyl carbazate (0.1 mol), and 4-methylbenzaldehyde (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.085 mol, yield 85%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93-0.97 Å; N—H = 0.86Å and with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C_{methyl}).

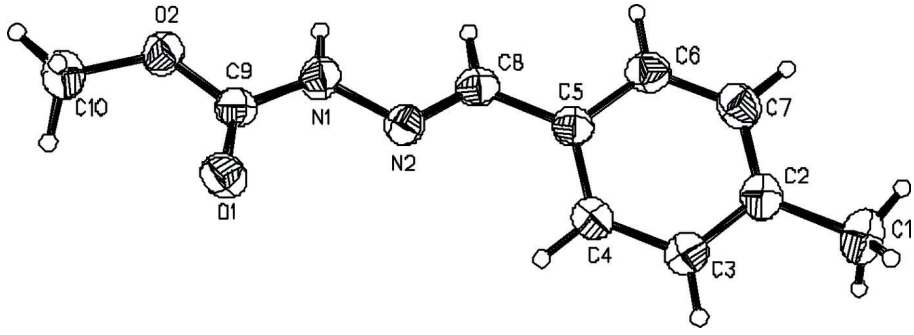

Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Methyl 3-(4-methylbenzylidene)carbazate

Crystal data

$C_{10}H_{12}N_2O_2$
M_r = 192.22
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.038 (2) Å
b = 13.308 (3) Å
c = 7.7923 (16) Å
β = 99.71 (3)°
V = 1026.1 (4) Å^3
Z = 4

$F(000) = 408$
$D_x = 1.244$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1798 reflections
$\theta = 3.5–25.2°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Blcok, colorless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
9493 measured reflections
2322 independent reflections
1528 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -13\rightarrow12$
$k = -17\rightarrow17$
$l = -9\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.175$
$S = 1.06$
2322 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1077P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O2	0.44060 (12)	0.82548 (9)	0.19195 (16)	0.0656 (4)
C9	0.55062 (16)	0.81674 (12)	0.1159 (2)	0.0531 (4)
N2	0.75692 (13)	0.73089 (10)	0.14969 (17)	0.0559 (4)
O1	0.56744 (12)	0.86340 (9)	−0.01208 (15)	0.0636 (4)
C5	0.95735 (16)	0.62998 (12)	0.1941 (2)	0.0540 (4)
N1	0.63541 (14)	0.74964 (11)	0.20247 (18)	0.0617 (4)
H1A	0.6141	0.7184	0.2906	0.074*
C8	0.82561 (17)	0.66004 (13)	0.2311 (2)	0.0580 (4)
H8A	0.7903	0.6261	0.3176	0.070*
C3	1.15344 (17)	0.65532 (14)	0.0599 (2)	0.0661 (5)
H3A	1.2004	0.6951	−0.0078	0.079*
C2	1.20961 (17)	0.56483 (14)	0.1252 (2)	0.0623 (5)

C7	1.13692 (18)	0.50763 (15)	0.2245 (2)	0.0683 (5)
H7A	1.1719	0.4465	0.2692	0.082*
C6	1.01357 (17)	0.53927 (13)	0.2585 (2)	0.0629 (5)
H6A	0.9668	0.4992	0.3260	0.076*
C4	1.02987 (18)	0.68754 (13)	0.0931 (2)	0.0647 (5)
H4A	0.9947	0.7484	0.0475	0.078*
C10	0.3380 (2)	0.89244 (15)	0.1047 (3)	0.0789 (6)
H10A	0.2630	0.8938	0.1665	0.118*
H10B	0.3080	0.8693	−0.0120	0.118*
H10C	0.3749	0.9589	0.1017	0.118*
C1	1.3469 (2)	0.53239 (18)	0.0914 (3)	0.0857 (6)
H1B	1.3699	0.4687	0.1463	0.128*
H1C	1.4131	0.5815	0.1383	0.128*
H1D	1.3449	0.5263	−0.0317	0.128*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0612 (7)	0.0707 (8)	0.0700 (8)	0.0078 (5)	0.0254 (6)	0.0082 (6)
C9	0.0607 (10)	0.0489 (8)	0.0527 (9)	−0.0029 (7)	0.0181 (8)	−0.0056 (7)
N2	0.0573 (8)	0.0566 (8)	0.0561 (8)	0.0004 (6)	0.0165 (6)	−0.0012 (6)
O1	0.0813 (9)	0.0577 (7)	0.0566 (7)	0.0059 (6)	0.0251 (6)	0.0061 (5)
C5	0.0561 (9)	0.0530 (9)	0.0517 (9)	−0.0046 (7)	0.0059 (7)	−0.0048 (7)
N1	0.0632 (9)	0.0686 (9)	0.0580 (9)	0.0076 (7)	0.0241 (7)	0.0119 (7)
C8	0.0613 (10)	0.0600 (10)	0.0535 (9)	−0.0063 (8)	0.0121 (8)	0.0016 (7)
C3	0.0633 (10)	0.0656 (11)	0.0719 (12)	−0.0027 (8)	0.0187 (9)	0.0010 (8)
C2	0.0575 (9)	0.0670 (11)	0.0597 (10)	0.0026 (8)	0.0025 (8)	−0.0105 (8)
C7	0.0672 (11)	0.0635 (11)	0.0698 (11)	0.0086 (8)	−0.0011 (9)	0.0068 (8)
C6	0.0629 (10)	0.0635 (10)	0.0606 (10)	−0.0035 (8)	0.0052 (8)	0.0096 (8)
C4	0.0675 (11)	0.0547 (9)	0.0736 (12)	0.0034 (8)	0.0169 (9)	0.0050 (8)
C10	0.0678 (12)	0.0721 (12)	0.0987 (15)	0.0134 (9)	0.0196 (11)	0.0115 (11)
C1	0.0677 (12)	0.1015 (15)	0.0883 (14)	0.0137 (11)	0.0145 (10)	−0.0081 (12)

Geometric parameters (Å, º)

O2—C9	1.343 (2)	C3—H3A	0.9300
O2—C10	1.442 (2)	C2—C7	1.379 (3)
C9—O1	1.2108 (19)	C2—C1	1.509 (3)
C9—N1	1.335 (2)	C7—C6	1.375 (2)
N2—C8	1.273 (2)	C7—H7A	0.9300
N2—N1	1.3742 (19)	C6—H6A	0.9300
C5—C4	1.389 (2)	C4—H4A	0.9300
C5—C6	1.390 (2)	C10—H10A	0.9600
C5—C8	1.456 (2)	C10—H10B	0.9600
N1—H1A	0.8600	C10—H10C	0.9600
C8—H8A	0.9300	C1—H1B	0.9600
C3—C4	1.378 (2)	C1—H1C	0.9600
C3—C2	1.389 (3)	C1—H1D	0.9600

C9—O2—C10	114.86 (13)	C6—C7—H7A	119.4
O1—C9—N1	126.40 (15)	C2—C7—H7A	119.4
O1—C9—O2	123.93 (15)	C7—C6—C5	121.34 (17)
N1—C9—O2	109.67 (14)	C7—C6—H6A	119.3
C8—N2—N1	114.77 (14)	C5—C6—H6A	119.3
C4—C5—C6	117.63 (16)	C3—C4—C5	120.62 (17)
C4—C5—C8	122.74 (16)	C3—C4—H4A	119.7
C6—C5—C8	119.61 (16)	C5—C4—H4A	119.7
C9—N1—N2	119.52 (13)	O2—C10—H10A	109.5
C9—N1—H1A	120.2	O2—C10—H10B	109.5
N2—N1—H1A	120.2	H10A—C10—H10B	109.5
N2—C8—C5	122.59 (16)	O2—C10—H10C	109.5
N2—C8—H8A	118.7	H10A—C10—H10C	109.5
C5—C8—H8A	118.7	H10B—C10—H10C	109.5
C4—C3—C2	121.59 (17)	C2—C1—H1B	109.5
C4—C3—H3A	119.2	C2—C1—H1C	109.5
C2—C3—H3A	119.2	H1B—C1—H1C	109.5
C7—C2—C3	117.61 (17)	C2—C1—H1D	109.5
C7—C2—C1	121.67 (17)	H1B—C1—H1D	109.5
C3—C2—C1	120.70 (18)	H1C—C1—H1D	109.5
C6—C7—C2	121.21 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.86	2.00	2.8615 (18)	176

Symmetry code: (i) x, $-y+3/2$, $z+1/2$.